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The Thermal Sensitivity of NF Compounds (U)

by

Jerome M. Rosen and Donald J. Glover

ABSTRACT: The systematic sensitivity difference between vicinally and geminally substituted bis(difluoroamino) alkanes previously reported by the Naval Ordnance Laboratory has been substantiated by measurements of additional compounds. For example, 1,2,4,5-tetrakis(difluoroamino)pentane initiated 180°C higher than the corresponding 2,2,4,4-isomer. Geminal NF₂ compounds with the substituents on an end carbon appear to fall in a different sensitivity class than compounds with the NF₂ groups on an internal carbon.

The most sensitive classes of NF₂ compounds proved to be the alkyl ethers containing the tris(difluoroamino)methyl moiety and the fluorinated biguanides.

Additional measurements are reported on a variety of compounds. (C)

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The sensitivity characteristics of several classes of difluoroamino organic compounds have been established. This information should provide guidance for the programs underway dealing with these very energetic materials.

The validity of the Wenograd method in assessing sensitivity has been clearly demonstrated. Numerous compounds shown to be sensitive by the Wenograd method proved to be sensitive in actual handling and by other methods of testing. Further, most of the NF₂ propellant formulation work is concerned with the class of compounds shown by the Naval Ordnance Laboratory to be the least sensitive, on an equal energy basis.

Work on this task was supported by the Advanced Research Projects Agency Order No. 23-61, Task 7, Item 2 under ONR Project NR 093-028, Sub-Project RR 001-06-02.

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The Thermal Sensitivity of NF Compounds (U)

An extensive program has been underway for a number of years for the synthesis of the difluoroamino (NF_2) class of organic compounds for high performance propellants. Numerous compounds have been made and as a class they are rather sensitive. The Naval Ordnance Laboratory is engaged in a study of the sensitivity characteristics of this class of energetic materials. Our goal is to provide information and guidance for the NF_2 program.

Thermal sensitivity measurements are reported on a number of interesting compounds. The results are discussed below.

Results and Discussion

We have continued to use the Wenograd thermal sensitivity method (1), (2) to characterize compounds, applying the same general techniques described in the previous study of difluoroamino compounds (3).

Illustrative of the data obtained is that for TAA and ATFE given in Table 3 and Figure 1. The straight line represents the least squares fit of the points.

These results and some of the others discussed were obtained on an improved apparatus, which features among other things a solid state power supply to replace storage batteries. This new apparatus gives a faster heating time, greater reproducibility, and reduced maintenance time. The improved reproducibility is shown by the generally smaller standard deviations obtained in recent measurements. We find only small differences in sensitivity between measurements made on the new and old equipment. For the most part, we would find no difficulty in intercomparing data for purposes of determining sensitivity relationships. A report is being prepared on the new apparatus (4).

When compounds are compared, the one requiring the highest temperature for thermal initiation at any given time delay is considered to be the least sensitive; likewise, the compound requiring the lowest temperature for thermal initiation at the corresponding time delay is the most sensitive. This interpretation is supported by Wenograd's work (5) in addition to more recent studies reported by Rosen, Holden and Glover (6).

For convenience, sensitivity comparisons are made using thermal initiation temperatures corresponding to delay times of 250 microseconds; the symbol, Temp₂₅₀, is used to represent the thermal initiation temperature at the 250 microsecond delay time.

In the first report on the sensitivity of difluoroamino compounds, we showed that there is a marked difference in thermal sensitivity between vicinally and geminally substituted bis(difluoroamino)alkanes (3). Similar measurements made on other vicinal and geminal isomer pairs confirmed the systematic difference between these two major classes of compounds, Table 1. At short delay times, the vicinal isomers required a somewhat higher temperature for thermal initiation than the geminal isomers, as shown by the Temp₂₅₀ data in Table 1.

The substantial sensitivity difference between vicinal and geminal compounds is demonstrated again by two isomeric tetrakis pentanes, Figure 2. In this case, the Temp₂₅₀ of 1,2,4,5-tetrakis(difluoroamino)pentane is 180°C higher than that of the 2,2,4,4-isomer.

Figure 3 is another rather interesting example of the relationship between structure and sensitivity. We can visualize the strong control of sensitivity by the geminal NF₂ grouping in 2,2-bis(difluoroamino)hexane. When this same structure contains and additional two NF₂ groups in the vicinal position, the energy is doubled but there is very little change in thermal sensitivity.

Previously we had pointed out the narrow range of Temp₂₅₀ values of the geminal class of hydrocarbons (3). This holds rather well for compounds containing up to six carbon atoms. However, with further dilution, a higher temperature for thermal initiation is required. This is shown by data obtained on 2,2-bis(difluoroamino)octane, Figure 4. Qualitatively, the geminal class behaves somewhat like nitroglycerine with added diluent (6). In the case of nitroglycerine, we find a relatively small increase in the thermal initiation temperatures until over 30% by weight of diluent is added.

Compounds with geminal NF₂ groups on a terminal carbon atom appear to form still another sensitivity class, Figure 5. Although we have limited data, the three compounds of Figure 5 show a different thermal sensitivity behavior from the geminal compounds listed in Table 1.

The geminal configuration on a terminal carbon atom is structurally similar to the vicinal class. In each case a carbon atom is bonded to hydrogen and an NF₂ group. The similarity in sensitivity behavior of these two classes would suggest a similarity in decomposition mechanism.

Location of the vicinal difluoroamino groups in the alcohol portion or the acid portion of an ester has very little effect on the thermal sensitivity. This is illustrated by the two isomeric butyrates in Table 3 and by comparing the propionate in Table 3 with its isomer, ethyl 2,3-bis(difluoroamino) propionate, previously reported (3).

Thermal sensitivity measurements were made on the following related tetrakis(difluoroamino) esters:

<u>Compounds</u>	<u>Code</u>
$ \begin{array}{ccccccc} \text{NF}_2 & \text{NF}_2 & \text{NF}_2 & \text{NF}_2 & & & \\ & & & & & & \\ \text{CH}_2 & -\text{CH} & -\text{CH} & -\text{CH} & -\text{CH}_2- & \text{O}- & \text{C}-\text{CH}=\text{CH}_2 \\ & & & & & & \\ & & & & & & \text{O} \end{array} $	Linear TAA
$ \begin{array}{ccccccc} \text{NF}_2 & \text{NF}_2 & & & \text{O} & & \\ & & & & & & \\ \text{CH}_2 & -\text{CH} & -\text{CH} & -\text{O}- & \text{C}- & \text{C}=\text{CH}_2 \\ & & & & & & \\ & & \text{CH}_2-\text{CH} & & & \text{CH}_3 & \\ & & & & & & \\ & & \text{NF}_2 & \text{NF}_2 & & & \end{array} $	TAMA
$ \begin{array}{ccccccc} \text{NF}_2 & \text{NF}_2 & & & \text{O} & & \\ & & & & & & \\ \text{CH}_2 & -\text{CH} & -\text{CH} & -\text{O}- & \text{C}- & \text{CH}=\text{CH}_2 \\ & & & & & & \\ & & \text{CH}_2-\text{CH} & & & & \\ & & & & & & \\ & & \text{NF}_2 & \text{NF}_2 & & & \end{array} $	TAA

As expected, the three compounds were found to be quite similar, Table 3. The Temp₂₅₀ values were only slightly higher than the corresponding value for 1,2,4,5-tetrakis(difluoroamino) pentane (3).

Six related alkyl ethers containing the tris(difluoroamino) methyl grouping were received for sensitivity measurements, Table 2. Three of the liquids fired in a few microseconds at a temperature in the range of 100 to 125°C. The other liquid,

FABE, exploded at the moment a sampling pipette touched its surface. Because of the ease with which these four liquids initiated, they should be considered as unusually sensitive.

Thermal sensitivities of the two solids listed in Table 2 are shown in Figure 6. FABDOL and FABDAN are slightly less sensitive than nitroglycerine by the Wenograd method.

In one attempt to load FABDOL, it initiated accidentally. After the sample tubes had been loaded they were being removed from the loading tube by means of a long metal hook. We believe one or two of the wires slipped off the hook and fell a short distance, perhaps 1 cm, into the remaining liquid sample. At this instant the sample exploded.

It is worth noting there is a distinct difference in sensitivity behavior between the liquid and solid compounds of Table 2. The differences in molecular structure are quite minor. Therefore we would not expect to find significant differences in sensitivity. About the only reason we can propose at present is that the liquids contain impurities which trigger their decomposition. Generally, liquids are more difficult to purify than solids. It is unfortunate that we have no information regarding the chemical quality of these interesting compounds.

Recent sensitivity studies carried out by the Esso Research and Engineering Company (7) show the high degree of sensitivity of the alkyl ethers containing the tris(difluoroamino)methyl grouping. There is good qualitative agreement between the Esso work and the Naval Ordnance Laboratory thermal sensitivity measurements.

The thermal sensitivity of an oxidant rich compound, FANG, is shown in Figure 5. Its structure is Y_3CNO_2 , where $Y = (NF_2)_3COCH_2$. FANG is sensitive by the Wenograd method as well as by the Naval Ordnance Laboratory impact machine, which shows it to be similar to lead azide.

An accidental explosion occurred with FANG. A long stainless steel rod had been dipped into the molten compound and then removed. This technique was used to remove a sample from the container. The sample, as received, was a solid mass on the bottom of a vial. It did not seem appropriate to dig it out with a spatula. The rod with solid adhering was placed vertically in a glass test tube. A second rod was dipped into the molten compound and removed. After solidification of the

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liquid the second rod was placed in the test tube. An explosion occurred when the ends of the rods touched at the bottom.

We experienced an accidental explosion with perfluoroguanidine. This took place after the hypodermic needle tubing had been filled with the sample. The filled sample tubes were being removed from the glass loading tube when the material exploded.

F

A sample of F_7BG , $F_2NC-N-C-NF_2$, exploded while the container was being opened.

A few compounds were received with a chromatographic purity less than 92%. These were considered too impure for our measurements.

There would appear to be little doubt that the fluorinated biguanide and the tris(difluoroamino)methyl types of compounds are unusually sensitive. Whether this sensitivity is the result of impurities or is inherent in the structure is not known. A recent study on the role of impurities on the impact sensitivity of perfluoroguanidine and compound "R", $FC(NF_2)_3$, shows the sensitizing effect of impurities (8). It is also concluded (8) these two compounds are impact sensitive, regardless of purity.

From the sensitivity viewpoint, the vicinal class of hydrocarbons appear to show the most promise for further developmental work. This is indicated not only by our measurements but by recent sensitivity studies of propellant formulations (9) carried out by the Rohm and Haas Company.

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Acknowledgement

Most of the compounds studies were supplied by the Reaction Motors Division of the Thiokol Chemical Corporation, under ARPA sponsorship. Some additional interesting structures were furnished by the Esso Research and Engineering Company and the Dow Chemical Company.

The late Francis Taylor, Jr. provided valuable assistance by measuring the purity of many of the compounds received for our study. We also acknowledge the assistance of Mr. Joshua D. Upton and Mr. George Swann.

TABLE 1
THERMAL SENSITIVITY OF BIS(DIFLUOROAMINO) ISOMERS

Vicinal Isomer	Temp., °C at 250 microsec. delay	Slope, $\frac{B}{a}$	Geminal Isomer	Temp., °C at 250 microsec. delay	Slope, $\frac{B}{a}$
1,2-propane	551	3.7	2,2-propane	502	7.7
1,2-pentane	664	3.5	2,2-pentane	489	6.5
2,3-pentane	670	4.0	3,3-pentane	481	6.9
1,2-hexane	765	3.8	2,2-hexane	502	8.3
2,3-bis(NF ₂)-3-methylpentane	677	4.8	3,3-hexane	507	5.7
1,2-bis(NF ₂)-2-methylpentane	704	4.7	2,2-bis(NF ₂)-4-methylpentane	502	5.9
1,2-bis(NF ₂)-3-methylpentane	709	4.7			
1,2-bis(NF ₂)-3,3-dimethylbutane	742	3.3			

$$\frac{1}{a} \log_{10} \text{ delay time in milliseconds} = A + \frac{1000B}{T, ^\circ K}$$

TABLE 2

THERMAL SENSITIVITY OF TRIS(DIFLUOROAMINO)METHYL ETHERS

Code	Structure	M. Pt., °C	Temp., °C at 250 microsec. delay
FAE	Y-CH ₃	Liquid	Exploded
FAB	Y-CH ₂ -CH ₂ -Y	Liquid	Initiates at 100-125°C
FABDE	Y-CH-CH-Y O	Liquid	Initiates at 100-125°C
FABDN	Y-CH-CH-Y O O NO ₂ NO ₂	Liquid	Initiates at 100-125°C
FABDOL	Y-CH-CH-Y OH OH	75	437
FABDAN	Y-CH-CH-Y OH O NO ₂	40	389
	Y=CH ₂ -O-C (NF ₂) ₃		

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TABLE 3
SUMMARY OF THERMAL SENSITIVITY DATA

Compound	Temp., °C at 250 microsec. delay	Slope B/a	Stand. Dev. of log ₁₀ delay time	No. of trials	Range milliseconds
1,1-Bis(difluoroamino)hexane	622	5.8	0.20	17	0.05 to 14.0
2,3- pentane	670	4.0	0.13	19	0.05 to 4.07
1,2- 3,3-dimethylbutane	742	3.3	0.18	25	0.06 to 13.7
3,3- hexane	507	5.7	0.19	12	0.03 to 18.8
2,2- octane	571	5.1	0.30	18	0.03 to 2.37
2,3- -2-methylpropionitrile	533	6.9	0.13	22	0.04 to 14.2
1,1- butane	537	8.7	0.22	24	0.01 to 21.6
1,1- propane	443	6.1	0.19	16	0.02 to 1.41
3,3- pentane	481	6.9	0.13	19	0.03 to 18.5
Di-[1,2,3-tris(difluoroamino)propyl ether, HPE	544	7.7	0.30	17	0.02 to 32.2
1,2,5-tetrakis(difluoroamino)hexane	493	7.6	0.18	18	0.06 to 37.4
1,2,4-tetrakis(difluoroamino)pentane	409	13.0	0.41	18	0.02 to 11.1
2-Methyl-1,2-bis(difluoroamino)ethyl acetate	748	4.0	0.13	20	0.07 to 5.57
1,2-Bis(difluoroamino)-2-methylpentane	704	4.7	0.10	20	0.06 to 5.67
2,3- -3- -3- ethane	675	4.8	0.12	22	0.05 to 6.55
1,2- ethane	709	4.7	0.12	19	0.04 to 5.56
Ethyl-2,3-bis(difluoroamino)butyrate	567	9.5	0.18	20	0.02 to 17.8
1,2-Bis(difluoroamino)ethyl propionate	716	3.5	0.22	16	0.08 to 2.57
1,2- butyrate	695	4.5	0.24	18	0.08 to 3.55
4,5- pentanoic acid	720	4.5	0.23	16	0.08 to 19.1
Y = -CH ₂ OC(NF ₂) ₃	653	3.3	0.13	22	0.04 to 52.4
Y-CH ₂ OCOCCH=CH ₂ , FAMEG	431	6.7	0.27	18	0.03 to 21.5
Y-CH-CH-Y, FABDAN ONO ₂ OH	389	3.7	0.15	14	0.05 to 5.97
Y-CH-CH-Y, FABDOL OH OH	437	5.5	0.12	9	0.05 to 1.83
Y ₃ C-NO ₂ , FANG	416	28.7	0.19	8	0.06 to 1.07
(NF ₂)CH ₂ (CHNF ₂) ₃ CH ₂ OCCCH=CH ₂ , linear TAA	619	8.0	0.26	19	0.04 to 2.09
(NF ₂)CH ₂ CHNF ₂) ₂ CHOCCCH=CH ₂ , TAA	603	6.0	0.15	19	0.02 to 7.27
(NF ₂)CH ₂ CHNF ₂) ₂ CHOCC-CH=CH ₂ , TAMA O CH ₃	626	6.2	0.20	19	0.03 to 6.55
CF ₃ CH ₂ O-C(NF ₂) ₂ -N, CATFE NF ₂ F	455	5.1	0.18	9	0.03 to 12.4
CF ₃ CH ₂ O-C(NF ₂) ₂ -N, ATFE NF ₂ F	360	11.5	0.24	12	0.03 to 3.87
1,1'-Bis(difluoroamino)dipropyl ether	686	6.1	0.23	19	0.07 to 3.66

$$\frac{1}{a} \log_{10} \text{delay time in milliseconds} = A + \frac{1000 B}{T, ^\circ K}$$

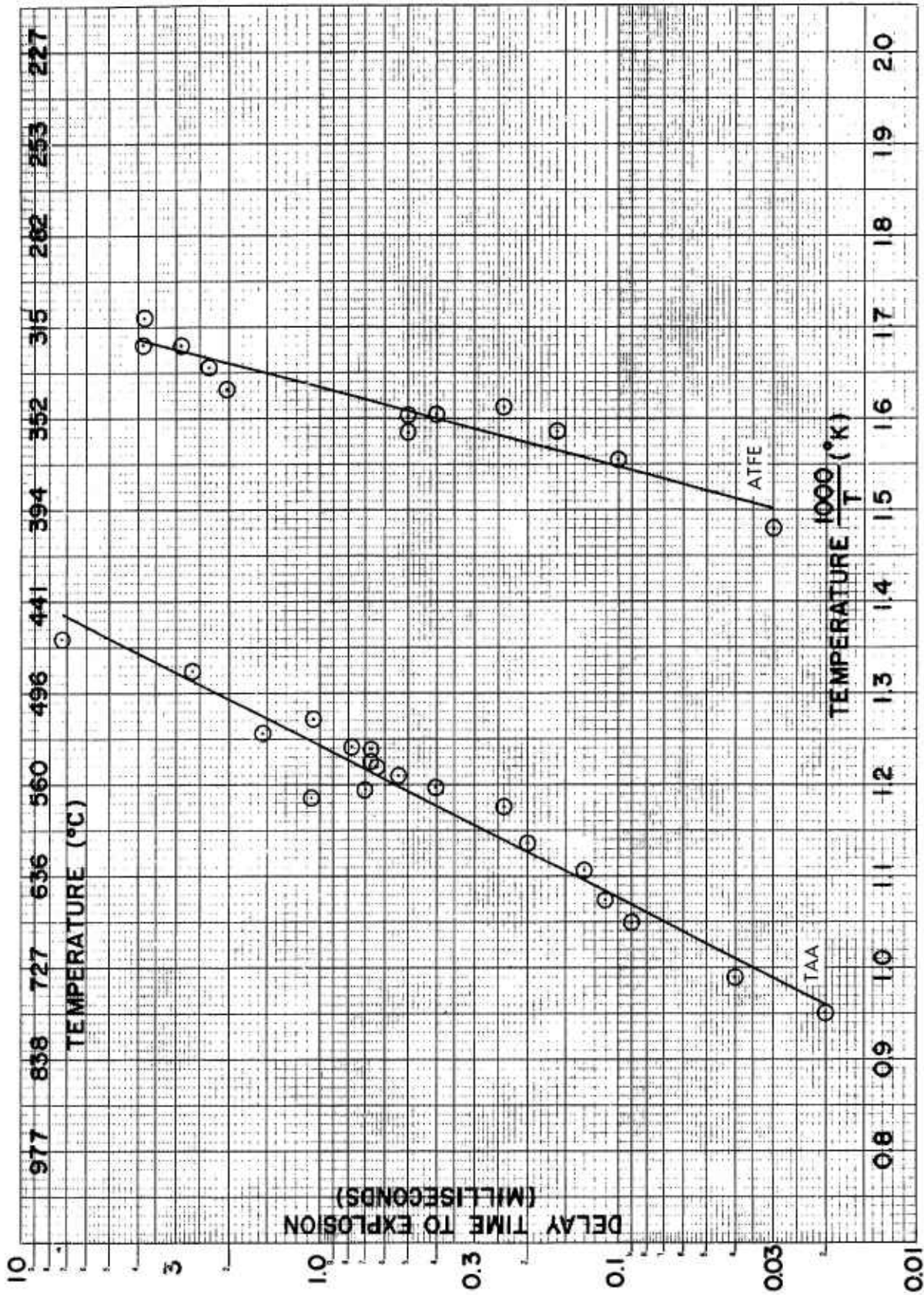


FIG. 1 DATA FOR TAA AND ATFE

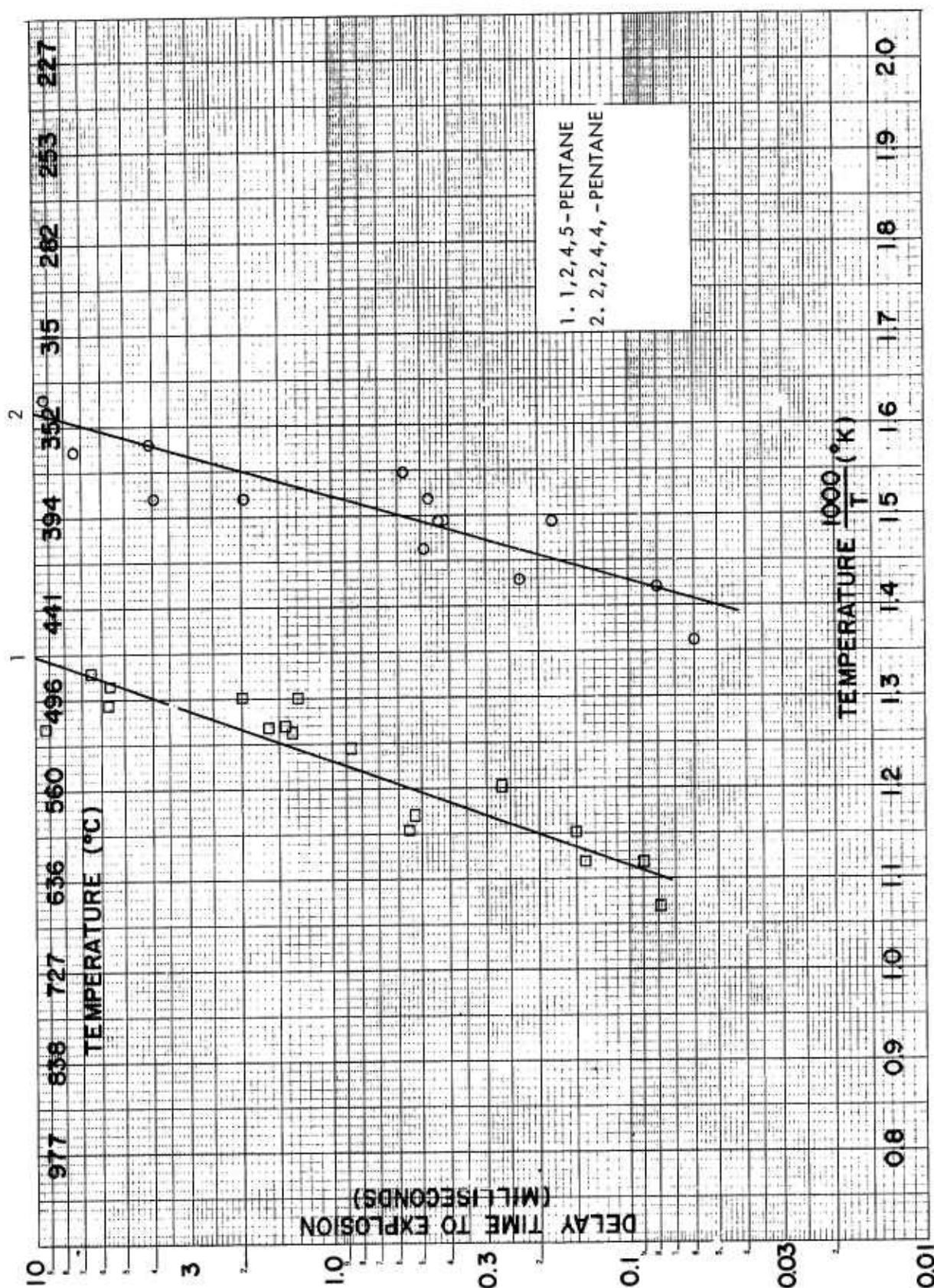


FIG. 2 THERMAL SENSITIVITY OF TETRAKIS DIFLUOROAMINO PENTANES

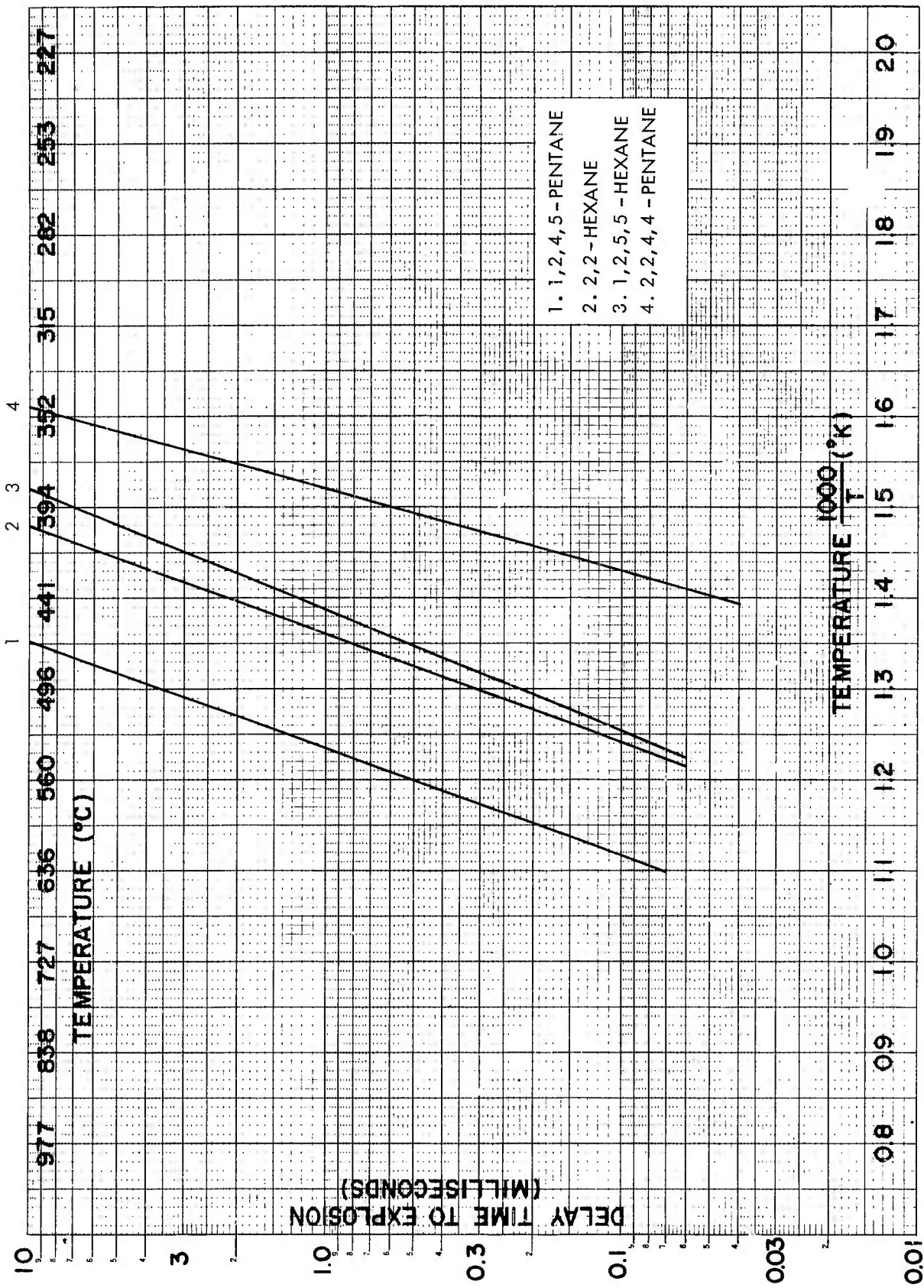


FIG. 3 VICINAL VS GEMINAL SENSITIVITY

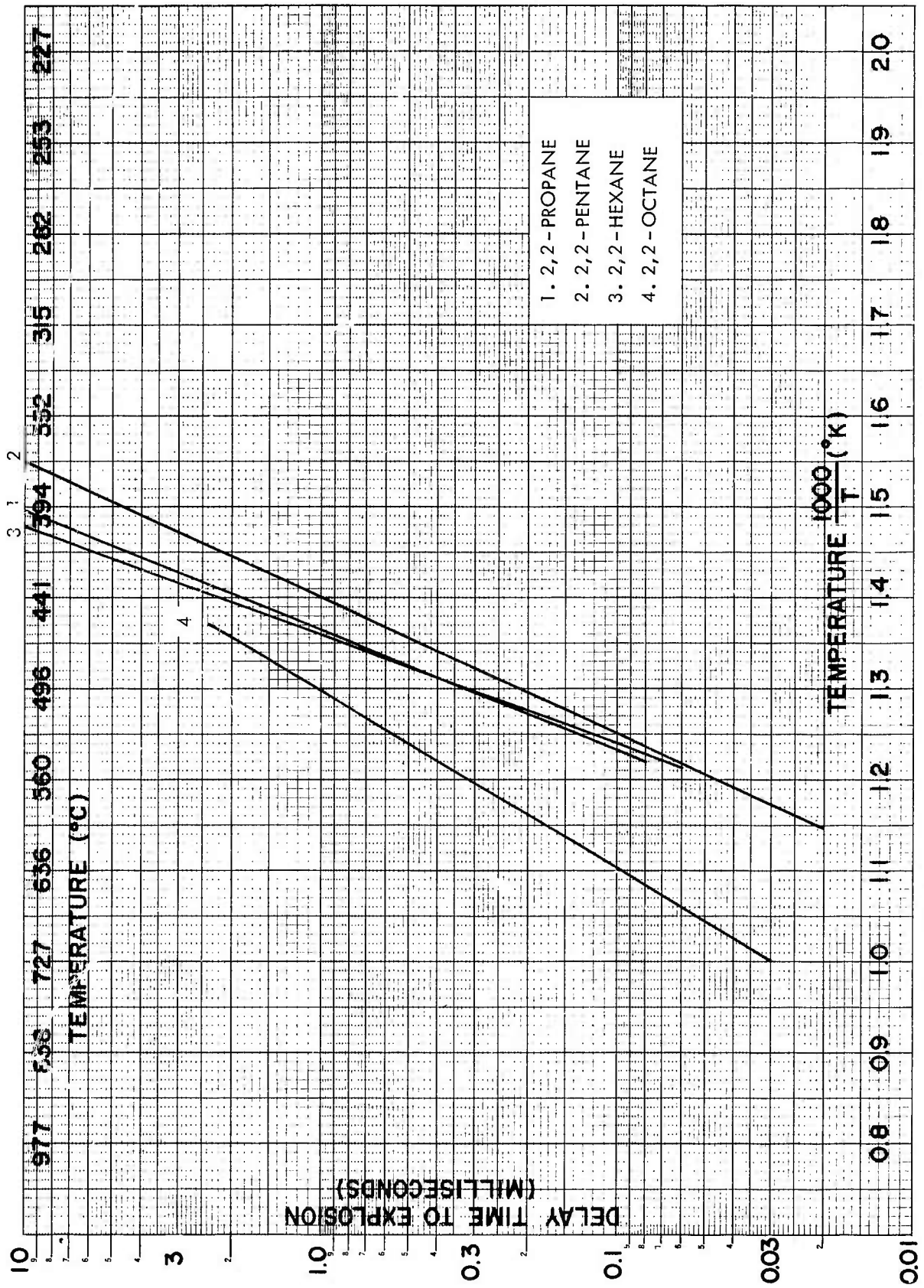


FIG. 4 THERMAL SENSITIVITY OF GEMINAL DIFLUOROAMINO COMPOUNDS

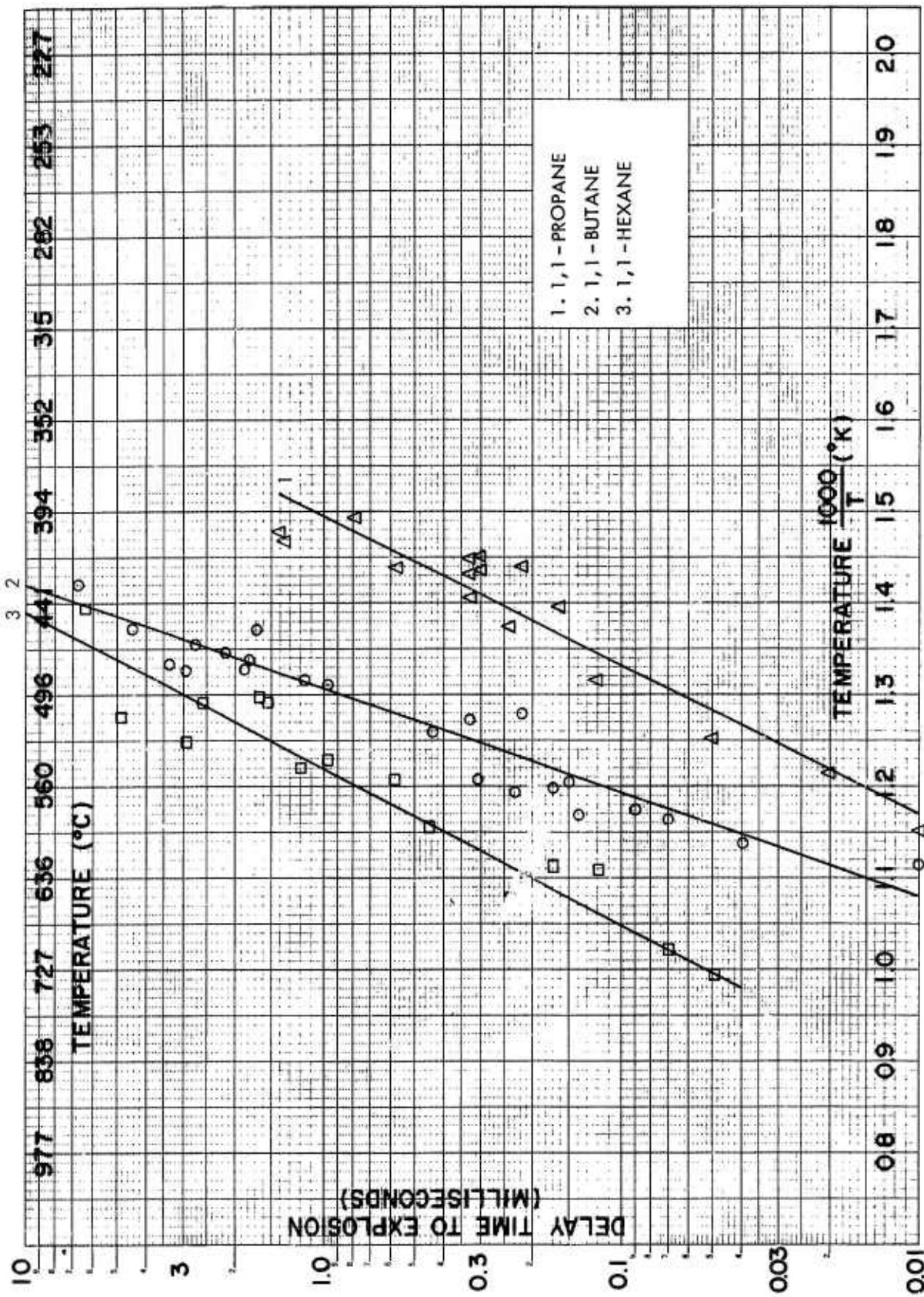


FIG. 5 GEMINAL NF_2 GROUPS ON TERMINAL CARBON ATOM

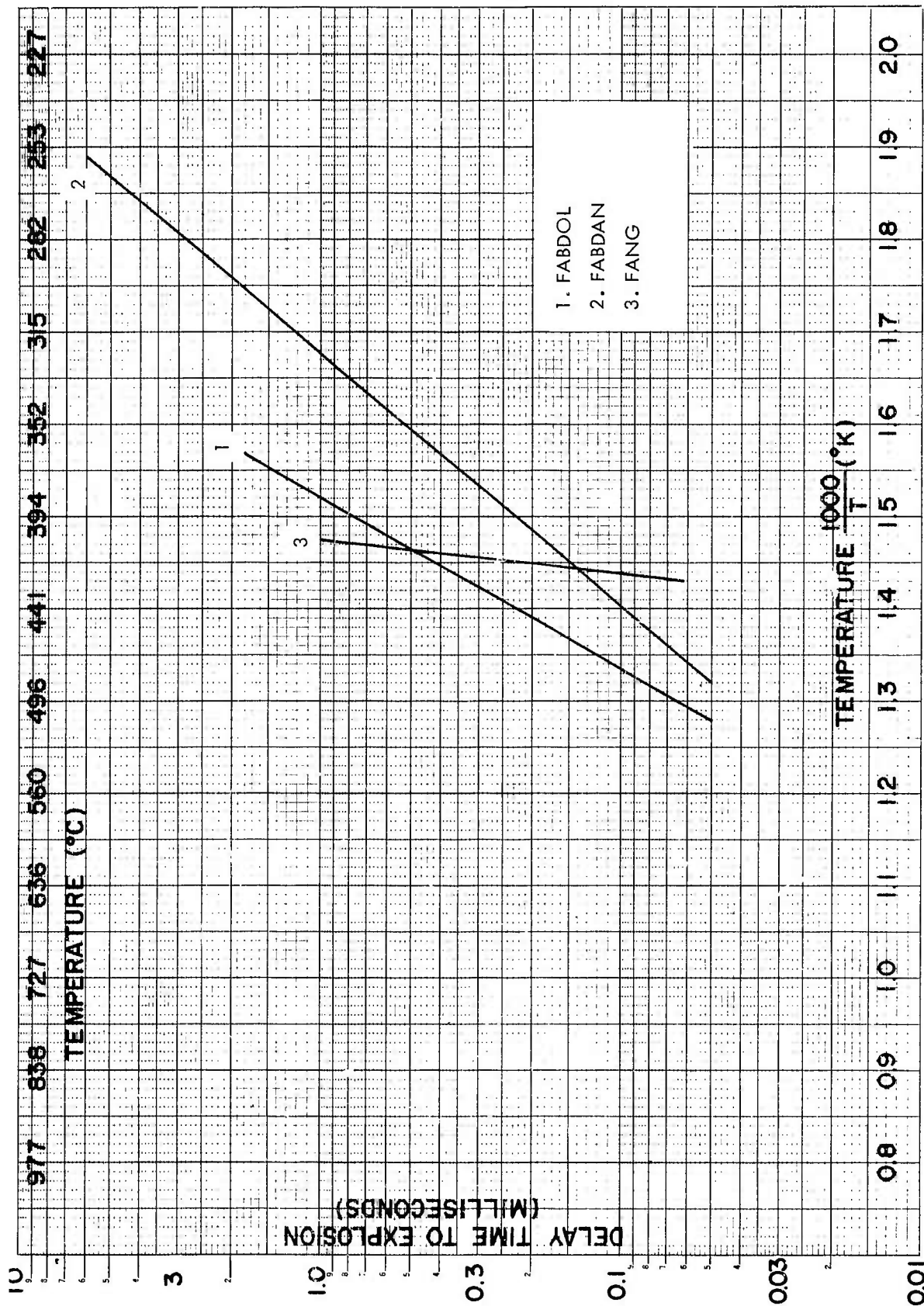


FIG. 6 TRIS(DIFLUOROAMINO) METHYL ETHERS

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<p>Naval Ordnance Laboratory, White Oak, Md. (NOL technical report 65-192) THE THERMAL SENSITIVITY OF NF COMPOUNDS (U), by Jerome M. Rosen and Donald J. Giever. 26 Nov. 1965. v.p. charts, tables. ONR project NR 093-028.</p> <p>CONFIDENTIAL</p> <p>Additional thermal sensitivity measurements are reported for a variety of bis(difluoroamine) compounds. The grouping of vicinally and geminally substituted compounds into separate classes reported previously has been substantiated. Still another class is formed by compounds geminally substituted on the terminal carbon atom. The most sensitive compounds are the fluorinated biguanides and the tris(difluoroamino)methyl ethers. (C)</p>	<p>1. Propellants Sensitivity</p> <p>2. Chemical compounds</p> <p>I. Title</p> <p>II. Rosen, Jerome M.</p> <p>III. Giever, Donald J.</p> <p>DOWNGRADED AT 3 YEAR INTERVALS DECLASSIFIED AFTER 12 YEARS DOD DIR 5200.10</p> <p>Abstract card is confidential</p>
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